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COMMENTARY ON U.S. ARMY STANDARD
TEST METHOD FOR FLEXURAL STRENGTH
OF HIGH PERFORMANCE CERAMICS AT
AMBIENT TEMPERATURE

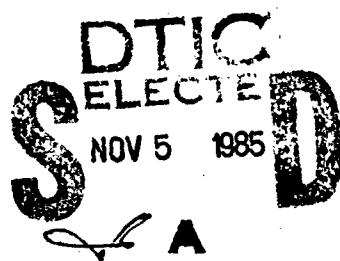
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→ This document → **ABSTRACT**

Military Standard 1942(MR), "Flexural Strength of High Performance Ceramics at Ambient Temperature," was developed as a standard test method which will reduce experimental error and will greatly enhance the reproducibility and consistency of strength data for Army systems. The standard is intended for, but not limited to, usage with monolithic ceramics such as alumina, silicon nitride, and silicon carbide. The value of flexural data for design is discussed in this report. Background information and considerations that led to the standard are presented.

metric System; bending jigs; error analysis
Keywords: mechanical properties;

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Ceramic materials

INTRODUCTION

The Army recognizes the potential applicability of the new ceramics to such systems as heat engines and missile radomes. One of the major obstacles to the use of ceramics as structural entities, devices, or component parts of military end items has been a definite lack of design data. The reliability of design data for ceramics is generally conceded to be of relatively low order as compared with such materials as metals and plastics. Part of this problem can be traced to the lack of standardization of test methods for high performance ceramics. In response to this need, MIL-STD-1942(MR), "Flexural Strength of High Performance Ceramics at Ambient Temperatures," dated 21 November 1983, has been developed. #1473

Strength is a fundamental property of interest to the designer. Due to the extreme cost and difficulty of conducting tension testing on ceramics, designers have resorted to using flexural strength data. Unfortunately there are no standard methods suitable for the high performance ceramics. As a result, the ceramics community in the United States currently uses a myriad of specimen sizes, fixture types, and testing procedures. There are serious problems with data compatibility and reproducibility, and some of the results contain serious experimental errors. This is particularly alarming considering that some of the error can be either systematic or random. Statistical analysis of strength data can be severely hampered if experimental errors are superimposed upon the material's inherent variability. These issues are clearly addressed in a National Material Advisory Board report:¹ "Use of statistics to account for a variability having an assigned cause, of course is quite different from normal usage of statistics in experimental work, and it introduces a peculiar problem - namely, that the ceramic strength data must be essentially free of experimental error.* If one reflects experimental error as well as flaw variability, the resulting statistical description of the ceramic will be incorrect ...and any stressed ceramic component designed on the basis of the description will perform unreliably." The report continued: "In general, insufficient attention is given to this need for error-free data in applying statistical fracture theory to design problems. . .In view of current practice in strength testing of ceramics, we think it possible that lack of adequate care in testing is a major cause of the unreliability problem being addressed here."*

In 1973, the Army Materials and Mechanics Research Center (AMMRC) formulated a proposed standard method.[†] This method was not adopted since it was determined that it contained serious faults. A detailed study was subsequently made of the experimental errors in flexure testing, culminating in the publication of a set of requirements to minimize such errors.^{2,3} The guidelines presented in these latter reports were used for the new standard.

The vast majority of flexural data is currently published without any mention of experimental error or specimen-fixture dimensional tolerances. Investigations have documented the existence and quantified the magnitude of errors in the past, and the reader is directed to References 2 and 3 as examples. Unfortunately, these

*Underline added for emphasis.

[†]Military Standard Test Methods for Structural Ceramics. Army Materials and Mechanics Research Center, 15 October 1973.

1. Reliability of Ceramics for Heat Engine Applications. National Materials Advisory Board NMAB-357, National Academy of Sciences, Washington, DC, 1980, AD D118 581.
2. BARATTA, F. I. Requirements for Flexure Testing of Brittle Materials. Army Materials and Mechanics Research Center, TR 82-20, April 1982, AD A113 937.
3. BARATTA, F. I., and QUINN, G. D. Errors Associated with Flexure Testing of Brittle Materials. Army Materials and Mechanics Research Center, to be published.

error analyses have largely been ignored. The most serious errors are usually due to external loading influences associated with the test jig including load bearing friction, bearing misalignment, bearing or specimen twist or wedging stresses.²⁻⁷ Some of the errors are calculated to be in the tens of percent and experimental evidence supports the analyses, e.g., References 4 and 7 cite frictional errors (due to use of rigid load bearing) of 14 and 13 percent, respectively. Frictional errors of this magnitude have been confirmed at AMMRC by experiments with alumina and silicon carbide. An extreme example is in Reference 8 which reports friction error of over 100 percent in coated glass rods.

Flexure strength testing is commonly used for quality control or material development. Experiments are performed to assess consistency among billets fabricated at the same time, or consistency as a function of time. Flexure testing is also valuable as a means to determine the strength-limiting flaws in a ceramic material. Portions of the Army standard method have been prepared with the above considerations in mind.

Although flexure tests are convenient to conduct, it is controversial whether the resulting data can be used as a source of design data. There are serious disadvantages associated with flexure testing. Flexural specimens experience a non-uniform stress state which exposes only a very small volume of material to the full tensile stress. Although there are statistical theories of strength that can be used to analyze the effect of specimen size on strength,^{3,4,9-11} extrapolation from small volume specimens to components that are many orders of magnitude larger may lead to inaccurate strength estimates. The confidence limits would broaden to the extent that safe design stresses would be extremely low. Flexural specimens are usually surface and edge sensitive. In many instances, strength measurements reflect only the machining damage incurred during specimen preparation. Such data could only be applied to predict failure of a component if it also failed from surface machining damage of the identical type. In reality this is usually not the case. Machined flexural specimens may be irrelevant with respect to components with as-fired surfaces. Flexural testing can also lead to inadvertent bias in the strength data. Some statistical theories of strength, as those proposed by Weibull,⁹ assume that each specimen contains a representative number of flaws,^{10,11} but in fact only a few could be contained within a small specimen size. Also, those specimens of very low strength can fracture during handling and machining, and thus are not included within the statistical sampling. An even more fundamental problem is that large flaws, such as large pores often found in sintered ceramic components, can be comparable in size to the flexural specimen cross section. Further complications arise during analysis when it is not certain which statistical distribution function is appropriate, i.e., whether it should be normal, lognormal, or Weibull.¹² For example, even when a Weibull analysis is appropriate, should a

4. STANLEY, P., SIVILL, A. D., and FESSLER, H. *The Unit Strength Concept in the Interpretation of Beam Test Results for Brittle Materials*. Proceedings of the Institution of Mechanical Engineers, Edmunds, Suffolk, UK, v. 190, 46/76, p. 585-595.
5. HOAGLAND, R. G., MARSCHALL, C. W., and DUCKWORTH, W. H. *Reduction of Errors in Ceramic Bend Tests*. J. Amer. Cer. Soc., v. 59, no. 5-6, May-June 1976, p. 189-192.
6. DUCKWORTH, W. H. *Precise Tensile Properties of Ceramic Bodies*. J. Amer. Cer. Soc., v. 34, no. 1, 1951, p. 1-9.
7. NEWNHAM, R. C. *Strength Tests for Brittle Materials*. Proceedings of the British Ceramic Society, no. 25, May 1975, p. 281-293.
8. RITTER, J. E., Jr., and WILSON, W. R. D. *Frictional Effects in Four-Point Bending*. Transactions of the American Society of Lubrication Engineers, v. 18, no. 2, 1974, p. 130-134.
9. WEIBULL, W. *Statistical Theory of Strength of Materials*. Royal Swedish Institute of Engineering Research, Proc. no. 151, 1939, p. 145.
10. DAVIES, D. G. S. *The Statistical Approach to Engineering Design Ceramics*. Proceedings of the British Ceramic Society, no. 22, 1973, p. 429-452.
11. JAYATILAKA, A. De S., and TRUSTRUM, K. J. J. Mat. Sci., v. 12, 1977, p. 1426-1430.
12. NEAL, D. M., and SPIRIDICLIOZZI, L. *An Efficient Method for Determining the "A" and "B" Design Allowables* in Proceedings of the 28th Conference on the Design of Experiments in Army Research Development and Testing, Army Research Office, Technical Report 83-2, 1983.

two or three parameter function be used; with a surface or volume function? It is obviously inappropriate to use flexure data, wherein specimens failed from machining damage, to predict failure of components when the strength-limiting flaws are inclusions or pores.

The answer to many of these difficulties is fractography. The proper interpretation of flexural data for design purposes requires a characterization of the strength-limiting flaws in both the flexural specimens and the final components. The flexural data can only be applied if the flaws are of the same type. It is routine practice at AMMRC to optically examine, at up to 80X, all flexural specimens. Representative ones are then viewed with the scanning electron microscope. The value of fractographic examination of flexure specimens is discussed at length by Rice¹³ who states: "The most significant experimental procedure that can aid the understanding of mechanical properties is a study of fracture surfaces, especially to identify fracture origins... It is indeed amazing the number of mechanical properties studies conducted that were extensively concerned directly or indirectly with the size and characters of flaws and microstructure from which failure originated in which no attempt was made to experimentally observe and verify the predicted or implied flaw character." It is beyond the scope of the Army standard test method to prescribe a subsequent fractographic examination, but such an examination is strongly recommended to enhance the value of the flexural data.

MIL-STD-1942(MR) has been prepared with these considerations in mind. Four-point loading is preferred to three-point loading because the volume of material under high stress is significantly greater in the former. Specific specimen and loading geometries are prescribed because strength can vary with size and the uncertainty regarding which statistical analysis is appropriate for a given material. Standard sizes will make data comparisons much easier. The actual sizes chosen, and the logic behind the choices are discussed in the following sections. Configurations were chosen to minimize experimental error which infests much of the data published to date.

SURVEY

A survey was performed to determine fixture types and specimen configurations currently employed for strength testing of high performance ceramics. This was done to assess the needs of the ceramic community and to determine what procedures are popular. The survey was not intended to be exhaustive, but to detect trends. One hundred thirty-seven types were found. In instances where an establishment had several identical fixtures, they were counted as one.

A list of the fixtures is shown in Table 1. Four-point loading is divided into several categories depending upon the size ratio of the inner to outer spans. The 1/4 four-point configuration has a ratio of 1:2 and 1/3 four-point configuration has a ratio of 1:3. Odd configurations are combined separately. Table 1 shows that four-point loading is more popular than three-point loading, but not overwhelmingly so. Three-point loading is commonly used by universities, private industry, the Japanese ceramic community, and, in general, by those not concerned with design data. The 1/4 four-point configuration is somewhat more popular than the 1/3 four-point configuration. A surprising number of odd combination fixtures are in use. The results were further analyzed in a manner such that each fixture was given a

13. RICE, R. W. *Microstructure Dependence of Mechanical Behavior of Ceramics* in Treatise on Materials Science and Technology, v. 11, Academic Press, NY, p. 199-381.

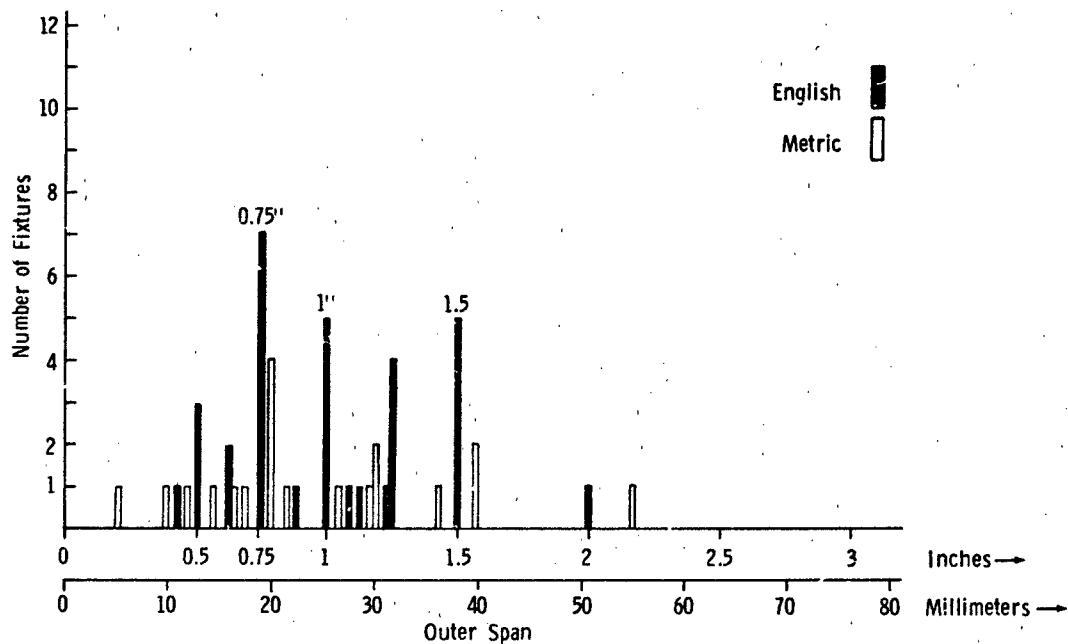
Table 1. NUMBER OF FIXTURE TYPES BY CATEGORY*

Establishment	Three-Point	1/4 Four-Point	1/3 Four-Point	Other Four-Point
U.S. Government	1	8	1	3
U.S. Private Institutions	1	3	1	3
U.S. Industry	15	9	8	9
U.S. Universities	9	2	6	5
Foreign	25	10	4	8
Totals	51	32	20	28

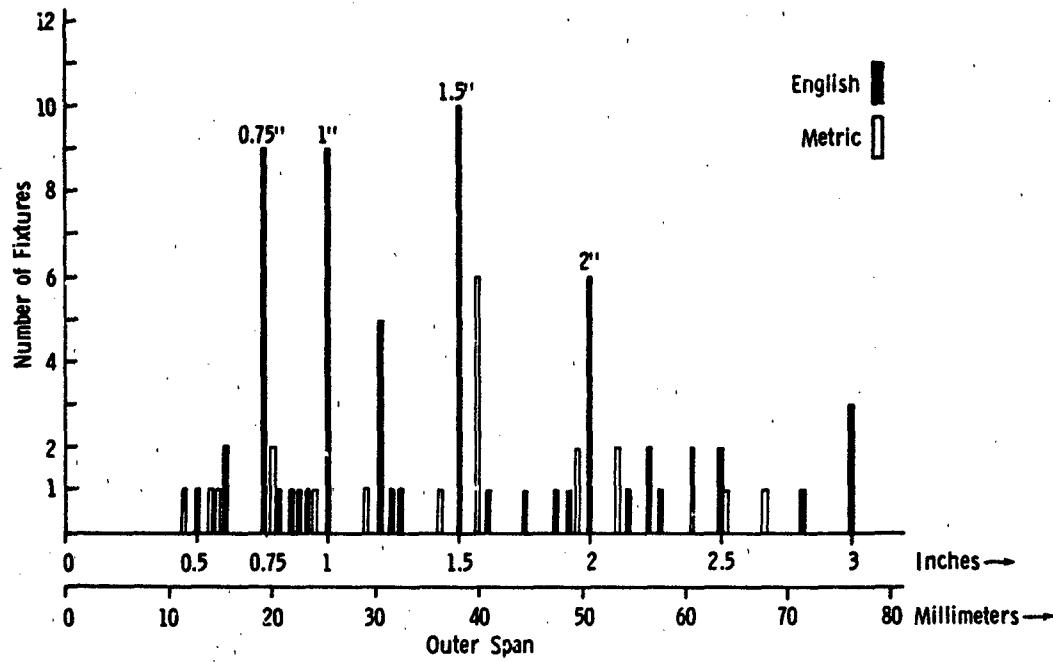
*Six other four-point fixtures were cited in literature, but span ratios were not given.

weighted value incorporating such consideration as its commercial availability, whether it is likely to be copied, the amount of data published, and whether the establishment had more than one fixture. In this manner, distinction could be made between a fixture used for years with much published data, and one used only for a few samples. The reformulation indicated a somewhat greater importance of the 1/4 four-point configuration relative to the others.

Figure 1 is a histogram for the outer spans of the three-point fixtures. Distinction is made between metric and English types. Most spans are in the 10 to 40-mm (0.5 to 1.5-inch) range. Although there are several peaks present, particularly at rounded numbers, there clearly is no dominant size. Figure 2 shows similar tabulations for the four-point fixtures. In this case, the spans are distributed over a wider range but, again, there is no dominant size. Many of the fixtures in the United States are used for material development purposes. Specimens



1. Histogram for the outer spans of the three point fixtures.



2. Histogram for the cutter spans of the four point fixtures.

are cut from research-sized billets which are often very small and of the order of 25 mm (1 inch) in size. Results of such testing are not favorable for design purposes however, due to the small volume of material under tension. Fixtures intended to generate design data are larger, of the order of 40 to 50-mm (1-1/2 to 2 inches) outer span. Even larger sizes are employed to test relatively weak materials (50 to 100 MPa strength). Thus, the survey indicates that no single fixture/specimen size will meet the needs of the high performance ceramics community. In addition, there is no strong preference as to size.

On the other hand, there are favored specimen cross-section sizes. Table 2 shows width to depth ratios in the instances where reported. Many establishments use square sections, or alternatively, rectangular sections that are twice as wide as thick. A number of establishments in the United States employ specimens with a 1/8-inch x 1/4-inch cross section. Unfortunately, there is no consistency with respect to the specimen lengths or fixture spans used in the latter groups. Also, only a few establishments in the United States employ metric fixtures.

Table 2. SPECIMEN CROSS-SECTION ASPECT RATIO REPORTED IN THE LITERATURE

Number of Fixtures	Width to Depth Ratio*					
	<1.0 0	1.0 29	Intermediate† 16	1.5 3	2.0 25	>2.0 15

*1.0 corresponds to a square, 2.0 is a rectangle such that the longest edge is parallel to the loading bearings.

^tBetween 1.0 and 1.5.

FOREIGN STANDARDS

Japan has a formal standard that is relevant: "JIS Testing Method for Flexural Strength (Modulus of Rupture) of High Performance Ceramics (R1601-1981)."^{*} Three- and four-point loading is permitted with an outer span of 30 mm. The inner span for four-point loading is 10 mm. The specimen size required is 3 mm x 4 mm x 30 mm. The standard has reasonable detail including specimen surface preparation and corner chamfer, and testing machine specifications. JIS R1601 has a commentary on the value of flexural testing, the applicability of the method to high temperature testing, and the influence of surface finish. The standard was formulated by eighteen of the most important ceramic establishments in Japan. In general, it is well organized, simple, and practical, however, it has shortcomings. Fixed knife edges are prescribed and although some concern is expressed about frictional effects, some experimental error will result. Surface preparation requirements are cited in the form of a final surface finish, which ignores probable subsurface machining damage. The 30-mm span is not optimal considering the needs of the United States community. It is too large for the developmental users who require a span less than 1 inch, yet too small for the design data community which favors 1-1/2 to 2-inch spans.

An informal standard has been developed by the German Institute for Research and Development for Air and Space Travel (DFVLR).[†] Only four-point testing is allowed, with spans of 20 and 40 mm. Specimen size is 3.5 mm x 4.5 mm x 45 mm. Very few details are stated regarding the fixturing, but significant comments are made pertaining to surface finishing. If as-fired, or sintered specimens are to be tested, then no finishing is necessary. Otherwise, the standard specifies a final surface finish and also requires complete documentation of the final step of preparation. The DFVLR method is prescribed in a letter only and has not been formally presented to the German Standards Committee. Nevertheless, the method is required of the firms that are engaged in the DFVLR program to develop ceramics for heat engines.

These foreign standards have been tailored to meet the needs of their respective nations. Neither standard is entirely satisfactory for the United States, e.g., one specimen size is not enough. The foreign standards are adequate for quality control or material development purposes, but have shortcomings with regard to generating design data.

U.S. ARMY STANDARD

MIL-STD-1942(MR) has been developed on the basis of the needs of the United States ceramics community, the requirements to minimize experimental error, a desire to metricate, and a view towards commonality with the foreign standards. The fixture and specimen sizes chosen are illustrated in Figure 3. Also shown for comparison are the German and Japanese spans and specimens. The three fixture!

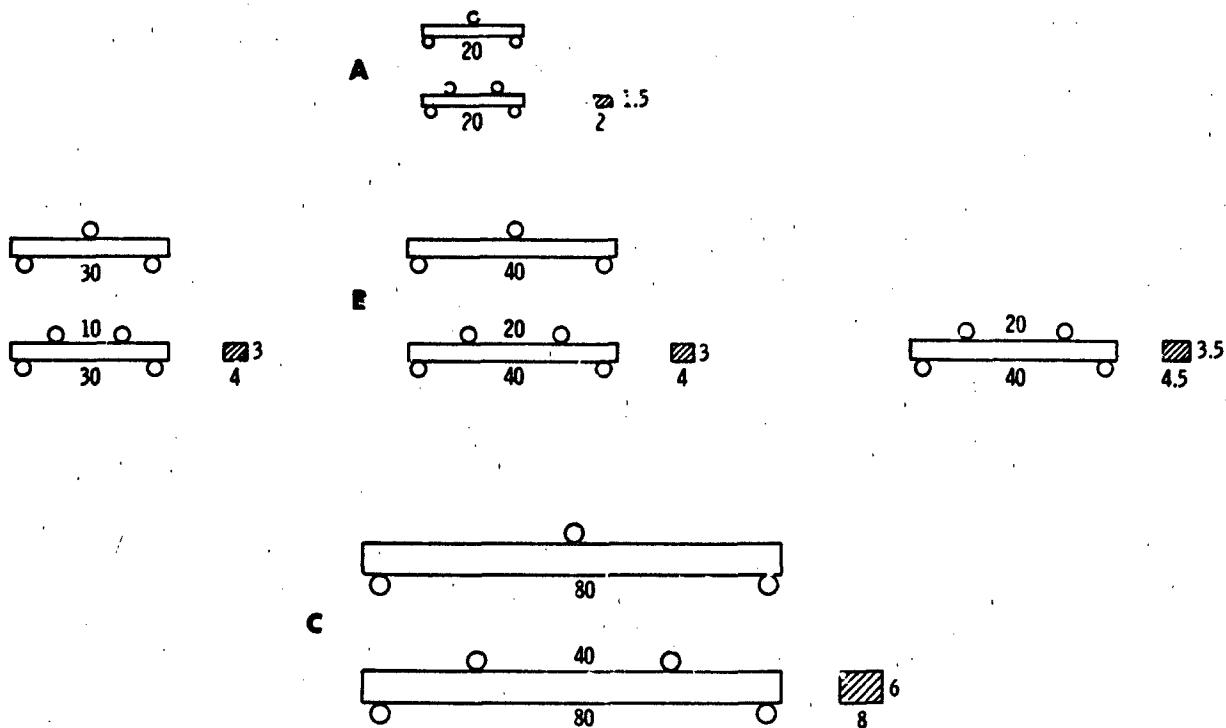
*Japanese Industrial Society. Original and English language copies are available upon request from authors.

†The standard is presented in a letter format (German language) from DFVLR to interested parties. The letter and an approximate translation are available upon request from authors.

JAPANESE STANDARD

US ARMY MIL STD 1942 (MR)

GERMAN STANDARD



3. MIL-STD-1942(MR) standard fixture and specimen sizes, with the Japanese and German standards shown for comparison. All dimensions are in millimeters.

specimen configurations of MIL-STD-1492(MR) are scaled to each other by a factor of two. The standard is all metric. Specimen A will satisfy the ceramic community which fabricates small billets. The size is not preferred for design data due to inherent experimental error (of the order of several percent for reasonably tight tolerances) and to the small volume of material under stress. Specimen B is preferred and should be eminently suitable for design data generation because of minimization of experimental error. The span for specimen B, 40 mm, should be satisfactory to a large contingent in the United States and is common with the German standard. Specimen C is suitable for weaker materials and for those seeking specimens with a larger volume under stress. The specimen cross-section dimensions are somewhat smaller than those ordinarily used in the United States. These dimensions have a profound effect upon the experimental errors that can arise.^{2,3} Many serious experimental errors arise from the use of too large a cross-section for a given fixture span. This is a problem with many of the fixture/specimen configurations in use in the United States today and can lead to errors greater than 5 percent. The cross-section chosen for the Army standard was carefully designed to keep experimental error minimized to less than a few percent. The adoption of the German specimen dimensions would have led to awkward dimensions for similarly scaled specimens A and C. The 3-mm x 4-mm size for the B specimen was finally chosen because it has low experimental error, is identical to the Japanese standard, and is similar to the German specimen.

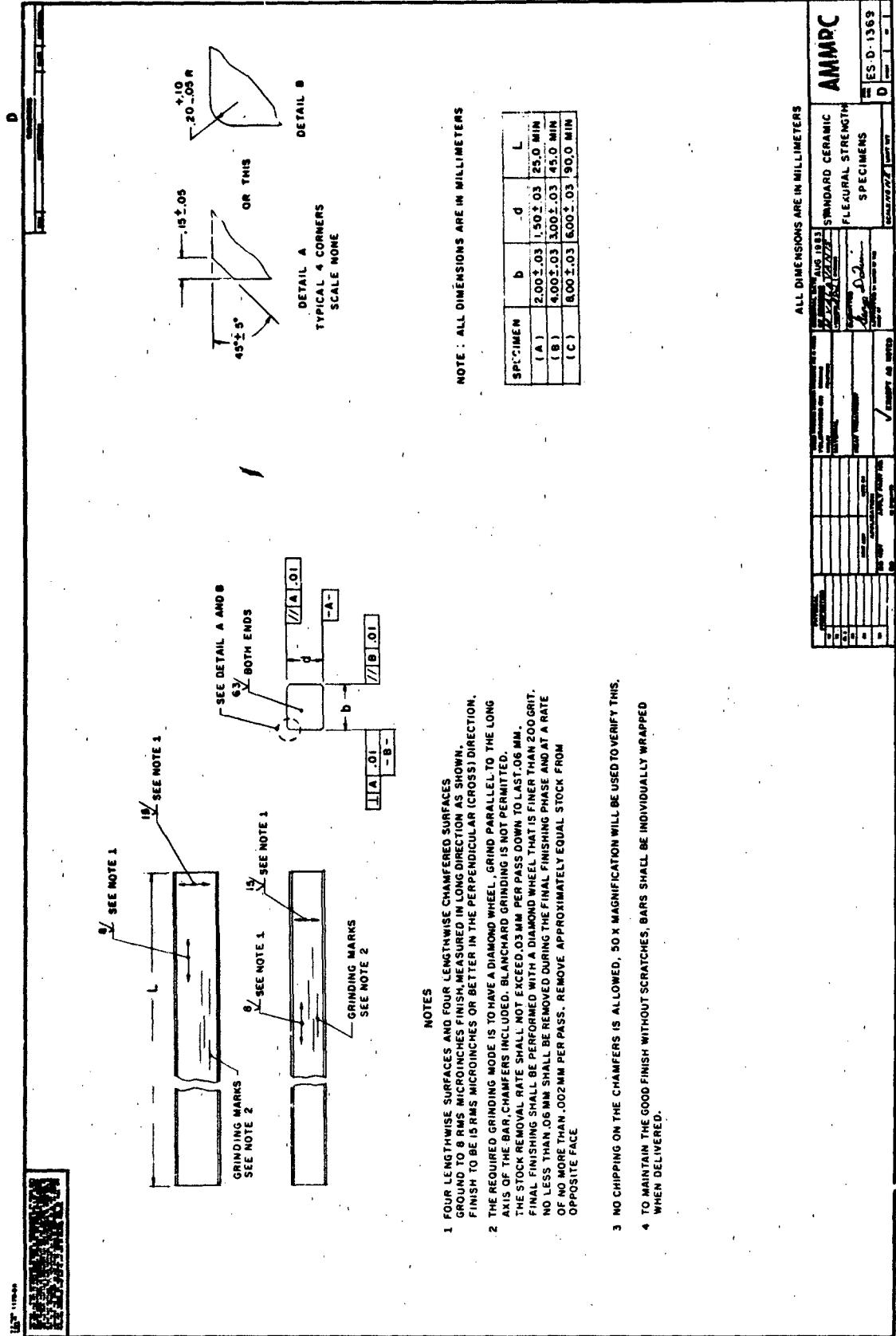
A 1/8-inch x 1/4-inch cross section was seriously considered for use with the 40-mm outer span fixture, but was deleted for several reasons. Twisting errors with common fixture and specimen tolerances were about 50 percent greater than the 3:4 cross-section. Metrication is a mandated Army policy for new standard preparation and the 1/8-inch x 1/4-inch specimen is redundant with the 3-mm x 4-mm specimen. Although a number of establishments are using the 1/8-inch x 1/4-inch cross-section specimen, the survey showed there is no consistency to the results since a wide range of fixture configurations and test procedures are employed. Indeed, there are far more establishments using other configurations in the United States, including square cross-section specimens.

A 2-mm x 4-mm cross-section specimen was also considered for the B configuration, especially since its experimental error is very low, but was not used for the following reasons: breaking loads (particularly for the scaled-down A configuration, could be quite low, causing practical problems. The stressed volume, or the amount of material under stress, is appreciably less than for the 3 mm x 4 mm specimen. Finally, it was dropped in the interest of eventual international standardization.

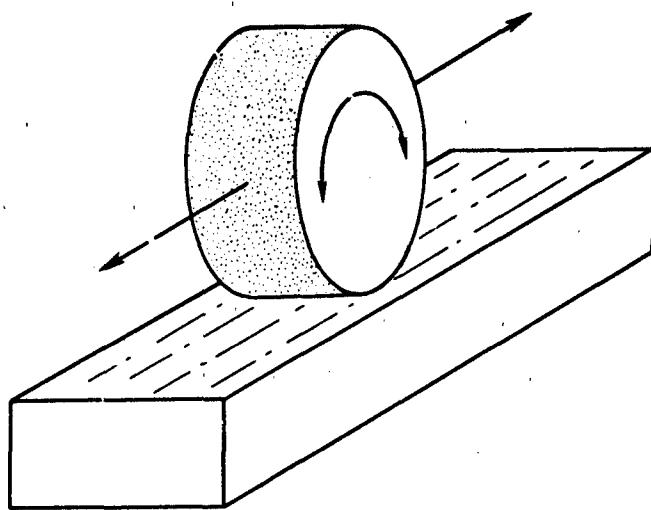
Three-point loading is permitted in the Army standard, but only for material development, quality control purposes, or to identify fracture origins in research studies.

Surface preparation can have a pronounced effect upon flexural strength. This occurs due to the introduction of machining related defects and the creation of residual surface stresses. In general, the stronger the ceramic, the more likely that machining damage can limit flexural strength. Specification of a final surface roughness is not adequate because machining damage can extend well below the surface striations. Lapping or polishing may remove surface striations and generate a perfect finish, but may not remove enough material to eliminate the much deeper machining damage. MIL-STD-1942(MR) allows three possibilities with respect to specifying specimen preparation. The first is the case wherein specimens with as-fired or as-fabricated surfaces are to be evaluated. Alternatively, it may be desired to reproduce the machining processes, on a bend specimen, that will be used for an actual component. In these two instances, no specification of surface preparation is given since it is not relevant. It is relevant if it is desired to ensure that specimens do not break in response to machining damage, or, finally, if it is intended to maintain a constant or reproducible level of machining damage. There are no standard machining procedures suitable for all high performance ceramics. MIL-STD-1942(MR) specifies a set of minimum requirements for specimen preparation (Figure 4). The standard calls for surface grinding only (as shown in Figure 5) and will not permit rotary or Blanchard grinding. All grinding shall be in the longitudinal direction. The wheel grits, speeds and rates of material removal specified are intended to eliminate or minimize severe machining damage or large residual stresses. As the standard evolves, or is modified in the future, specimen preparation may become more stringent.

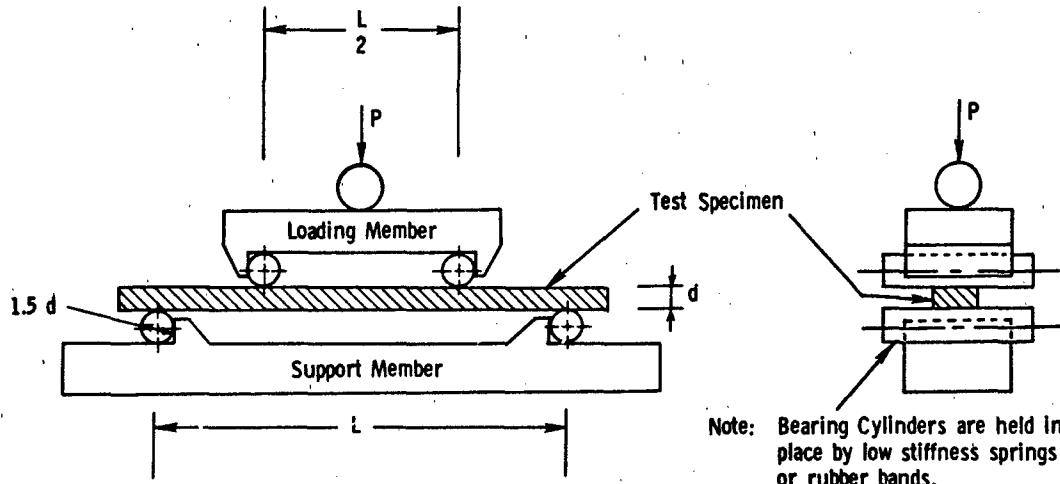
Fixture requirements are simple in order to be inexpensive and practical, but stringent in certain tolerances so as to minimize experimental error. If specimens can be prepared according to specifications (particularly with respect to curvature, parallelism and twist), then a simple four-point fixture as shown schematically in Figure 6 is permitted. The upper (or lower) loading member must be allowed to rotate to ensure that there is equal distribution of load. The upper bearings (inner span) must be carefully aligned relative to the lower bearings (outer span)



4. Machining requirements of MIL-STD-1942(MR) for specimens that are not as-fired or sintered, or machined to match a specific application.



5. The surface grinding method of surface preparation.



6. A schematic showing a suitable four-point fixture.

and the spans themselves must be accurate to within 0.1 mm. Of special interest is that the bearings must be free to rotate in order to eliminate friction error. These bearings need not be held by special equipment and can rest and roll on a flat surface. The shoulders are recommended to hold the bearings at the prescribed distances. The bearings themselves shall have a diameter of approximately 1.5 times the specimen thickness. They can be made of steel or other material with an elastic modulus no less than 2×10^5 MPa ($\sim 30 \times 10^6$ psi), and with a hardness no less than HRC 40. The bearings, the loading and support members should be no less than three times wider than the specimen width because of free surface end effects in the support. This will ensure against permanent fixture deformation for a specimen with a modulus as high as 5×10^5 MPa (70×10^6 psi) and a strength as high as 1400 MPa (200×10^3 psi).

If a specimen is warped, twisted, or cannot meet the parallelism requirements, then a more elaborate fixture is necessary. Such a fixture is described in Reference 5.

Ten specimens are required for estimates of the mean fracture strength. A minimum of 30 specimens are necessary if other statistical parameters (such as Weibull moduli) are to be estimated.

Crosshead rates vary for the three-test configurations. They are set at 0.2, 0.5 and 1.0 mm/min respectively for the A, B, and C arrangements. These will yield strain rates of the order of 1.0×10^{-4} in each case (irrespective of which loading system is used). The speeds were chosen to permit fast and practical specimen failure times and to minimize the time available for stress corrosion phenomena. The Japanese and German standards prescribe a crosshead rate of 0.5 mm/min for their specimens which is comparable to the MIL-STD-1942(MR) specimen B.

REVIEW PROCEDURE

The original draft version of MIL-STD-1942(MR) was prepared in late 1982. This draft, and several subsequent revisions, was distributed to over 250 scientists and engineers across the United States, Japan and Germany. Three oral presentations at technical meetings across the United States were given. As a result of this exposure dozens of critiques were received and extensive revisions were made. MIL-STD-1942(MR) contains major changes to, and supersedes all earlier draft revisions. One notable change is that there is an extensive introduction to the standard which discusses the value of flexure data, particularly for design data generation. A similar discussion exists in JIS 1601. This was done primarily in response to repeated requests by the United States technical community for such, and to eliminate any chance the standard could be misused. After a lengthy review process of one year, the standard was accepted and formalized by a committee at AMMRC in late 1983.

CONCLUSION

The Army standard method MIL-STD-1942(MR) is suitable for quality control, material development, and design data generation. Three specimen fixture sizes are prescribed since no one single size will meet the needs of the United States high performance ceramic community.

The four-point specimen/fixture combinations B and C are preferred for design data generation. Fractography is strongly recommended for design data interpretation. It is possible that fractography may become mandatory in future revisions.

In the spring of 1984, AMMRC began to perform, wherever possible, all flexural testing at ambient temperature on high performance ceramics according to MIL-STD-1942(MR). Additional testing on a trial basis is being performed at elevated temperature as well. It is not AMMRC's intent to mandate testing according to the standard at this time, but rather to lead by example. Specimen and testing jig blueprints are available upon request from the authors.

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Army Materials and Mechanics Research Center,
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COMMENTARY ON U.S. STANDARD TEST METHOD FOR
FLEXURAL STRENGTH OF HIGH PERFORMANCE
CERAMICS AT AMBIENT TEMPERATURE
George D. Quinn, Francis I. Baratta and
James A. Conway
Technical Report AMMRC TR 85-21, August 1985, 14 pp -
illus-tables, AMMRS Code 728012.12

Military Standard 1942(MR), "Flexural Strength of High Performance Ceramics at Ambient Temperature," was developed as a standard test method. The standard will reduce experimental error and will greatly enhance the reproducibility and consistency of strength data for Army systems. The standard is intended for, but not limited to, usage with monolithic ceramics such as alumina, silicon nitride, and silicon carbide. The value of flexural data for design is discussed in this report. Background information and considerations that led to the standard are presented.

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